(1) Publication number:

0 131 393

A1

G

#### **EUROPEAN PATENT APPLICATION**

(21) Application number: 84303972.8

(a) Int. Cl.4: C 11 D 3/20 D 06 M 13/16

(22) Date of filing: 13.06.84

30 Priority: 15.06.83 US 504648

Date of publication of application: 16.01.85 Bulletin 85/3

(94) Designated Contracting States: BE DE FR GB IT NL (7) Applicant: THE PROCTER & GAMBLE COMPANY 301 East Sixth Street Cincinnati Ohio 45201(US)

(7) Inventor: Huntington, Gregory Bruce 8593 Denailen Drive Cincinnati Ohio 45230(US)

(2) Representative: Gibson, Tony Nicholas et al, Procter & Gamble (NTC) Limited Whitley Road Longbenton Newcastle upon Tyne NE12 9TS(GB)

(64) Glyceryl monoester emulsifier systems for improved oily soil removal in detergent compositions.

(5) An emulsifier system comprising monolaurin and monocaprin which provides enhanced grease and oily soil removal in laundering fabrics, especially at low washing temperatures; and detergent compositions containing said emulsifier system.

EP 0 131 393 A1

# GLYCERYL MONOESTER EMULSIFIER SYSTEMS FOR IMPROVED OILY SOIL REMOVAL IN DETERGENT COMPOSITIONS Gregory B. Huntington

# FIELD OF THE INVENTION

This invention relates to detergent compositions and wash solutions and, in particular to a specific emulsifier system for use in said compositions and solutions to provide enhanced grease and oily soil removal from fabrics, even at relatively low washing temperatures.

## BACKGROUND OF THE INVENTION

The formulation of cleaning compositions with mixtures of surfactants in order to obtain improved detergency or controlled suds formation is well known in the detergent art. More especially the blending of different surfactant types for this purpose is well understood and numerous mixtures e.g., of nonionic and anionic or zwitterionic surfactants or of anionic, nonionic and cationic surfactants have been disclosed. Typical examples of mixtures of the binary type are described in U.S. Pat. No. 4,102,823, Matheson et al., issued January 25, 1978, and U.S. Pat. No. 3,920,569, issued November 18, 1975; and examples of ternary mixtures are provided by U.S. Pat. No. 4,321,165, Smith et al., issued March 23, 1982, U.S. Pat. No. 3,915,882, Nirschl et al., issued October 28, 1975, and British Pat. Nos. 873,214, Brunt et al., published July 19, 1961, and 614,297, General Aniline and Film, published August 9, 1950.

It has now been found that certain glyceryl monoesters, specifically the glyceryl monoesters of  $C_{10}$  and  $C_{12}$  aliphatic carboxylic acids, when used in combination with each other in detergent solutions containing conventional surfactants, can provide improved removal of oily soils from said fabrics, even at the lower washing temperatures which are tending to become common in household laundering.

Glyceryl monoesters of fatty acids are surfactants employed primarily as emulsifiers in the foods industry but their low solubility in water has generally tended to discourage their use in

5

10

20

25

30

35

THE COMPANY OF A PARTY THE PARTY THE

detengent compositions designed for use in aqueous media. British Pat. No. 1,564,507, Unilever, published April 10, 1980, discloses aqueous bath foam compositions comprising from 1 to 50% by weight of glyceryl monoesters of  $C_8^-$ C $_{14}^-$  fatty acids with from 5% to 50% by weight of anionic surfactants. The compositions are intended for personal cleansing (e.g., bath and shower) use and for washing of dishes and fabrics. The patent emphasizes low solubility of the fatty monoglycerides as facilitating their deposition on skin. The intended function of the monoglycerides in the said compositions is as skin emollient, rather than as an adjunct to detergency.

Japanese Patent Application No. 73500/73, Ajinomoto, Inc., published March 11, 1975, discloses a liquid detergent composition comprising an N-long chain acylated amino acid salt and a fatty acid nonlonic surfactant, which can be glyceryl monostearate, optionally in the presence of a sequestering agent such as EDTA, NTA or citrate. Here, also, the emphasis is on the function of the monoglyceride as a skin conditioning agent.

U.S. Pat. No. 4,192,761, Peltre et al., issued March 11, 1980 teaches the use of glyceryl fatty esters containing at least 16 carbon atoms as components of suds suppressing systems for detergent compositions, including anionic and nonionic surfactants.

Glyceryl monoesters of C<sub>12</sub>-C<sub>18</sub> fatty acids have also been disclosed as components of dryer-added sheet substrate fabric softening compositions, examples of such disclosures being those in U.S. Pat. No. 4,000,340, Murphy et al., issued December 28, 1976; U.S. Pat. No. 3,632,396, Perez-Zamora, issued January 4, 1972; and British Pat. No. 1,571,527, Norris, published July 16, 1980.

It is the object of the present invention to provide emulsifier compositions for laundry use which improve the oily soil removal capabilities of said compositions in aqueous solution, particularly under low temperature laundering conditions.

35

5

10

15

20

25

30

The office depositions and selection of the selection of

### SUMMARY OF THE INVENTION

The invention is directed to a emulsifier system for improving the oily soil removal properties of laundry detergents, the said emulsifier system comprising a mixture of glyceryl monolaurin:glyceryl monocaprin in a weight ratio of 80:20 to 70:30.

# DETAILED DESCRIPTION OF THE INVENTION

Broadly, the present invention comprises a emulsifier system comprising a mixture of glyceryl monolaurate:glyceryl monocaprate in a weight ratio of 80:20 to 70:30. This emulsifier system is especially effective in improving the oily soil removal performance of conventional laundry detergents at low wash temperatures, e.g., 60°-95°F (15°-35°C). Neither component alone is as effective as the combination at these low temperatures.

The invention also comprises detergent compositions comprising from 5% to 60% of an anionic or ethoxylated nonionic surfactant from about 5% to about 60% of the emulsifier system and from 0% to 60% of a sequestering builder.

All ratios and percentages herein are "by weight" unless otherwise stated.

# The Emulsifier System

The emulsifier system of the present invention comprises a mixture of monolaurin:monocaprin in a weight ratio of 80:20 to 50:50, preferably about 80:20 to 70:30.

The glyceryl esters useful in the present invention are the monoesters of  $C_{10}$  and  $C_{12}$  aliphatic carboxylic acids, i.e., capric and lauric acid. The carboxylic acids may be derived from natural sources i.e., vegetable oils or animal fats, or may be synthetic in origin i.e., from olefin build-up or Oxo synthesis.

The preparation of glyceryl monoesters is well known in the art, and is conventionally carried out by means of a two stage process involving partial glycerolysis of triglyceride, followed by molecular distillation. Although it is preferred that the monoester comprises at least 90%, most preferably at least 95% by weight of the glyceryl ester component, esters of monoester content of 70%

۲,

.

ř

5

10

15

20

25

30

can be used in which the major portion of the remainder is usually the diester together with minor proportions of glycerin and fatty acid. The level of tri ester (i.e., the starting triglyceride) should be minimized and should not exceed 1% by weight of the ester component, as it constitutes a cleaning load. Separation of the desired monoester or monoester fraction from the glycerolysis reaction mixture is normally carried out by means of vacuum distillation.

In the case of the glyceryl monocaprate, it may be more convenient to esterify the glycerin with capric acid. This technique also has to be used where synthetically derived fatty acids are employed in the monoester synthesis.

In order to provide the grease and oily soil removal benefits obtainable with detergent compositions in accordance with the invention, the detergent composition should contain from 5% to 60% (preferably from 20% to 45%, most preferably from 25% to 35%) of the monolaurin:monocaprin emulsifier system.

The amount of a composition added to a laundering solution should be such as to produce a concentration of from 0.005% to 0.25%, preferably from 0.01% to 0.015% of the emulsifier system in the solution.

#### Surfactants

Surfactants comprise from 5% to 60%, preferably from 25% to 50%, most preferably from 35% to 45% of the detergent compositions herein. Preferably the surfactants are anionic, but nonionics can be used, either alone or in combination with anionics.

The anionic surfactant can be any one or more of the materials used conventionally in laundry detergents, and include fatty acid soaps such as sodium or potassium salts of  $C_8$  to  $C_{20}$  fatty acids as well as synthetic anionic surfactants. Suitable synthetic anionic surfactants are water-soluble salts of alkyl benzene sulfonates, alkyl sulfates, alkyl polyethoxy ether sulfates, paraffin sulfonates, alpha-olefin sulfonates, alpha-sulpho-carboxylates and

7673

5

10

15

20

25

their esters, alkyl glyceryl ether sulfonates, fatty acid monoglyceride sulfates and sulfonates, alkyl phenol polyethoxy ether sulfates, 2-acyloxy alkane-1-sulfonates, and beta-alkyloxy alkane sulfonates.

A particularly suitable class of anionic surfactants includes water-soluble salts, particularly the alkali metal, and alkanolammonium salts of organic sulfuric reaction products having in their molecular structure an alkyl or alkaryl group containing from 8 to 22, especially from 10 to 20 carbon atoms and a sulfonic acid or sulfuric acid ester group. (Included in the term."alkyl" is the alkyl portion of acyl groups). Examples of this group of synthetic detergents which form part of the detergent compositions of the present invention are the sodium and potassium alkyl sulfates, especially those obtained by sulfating the higher alcohols (C8-C18) carbon atoms produced by reducing the glycerides of tallow or coconut oil and sodium and potassium alkyl benzene sulfonates, in which the alkyl group contains from 9 to 15, especially 11 to 13, carbon atoms, in straight chain or branched chain configuration, e.g., those of the type described in U.S. Pat Nos. 2,220,099 and 2,477,383 and those prepared from alkylbenzenes obtained by alkylation with straight chain chloroparaffins (using aluminum trichloride catalysis) or straight chain olefins (using Especially valuable are linear hydrogen fluoride catalysis). straight chain alkyl benzene sulfonates in which the average of the alkyl group is 11.8 carbon atoms, abbreviated as  $C_{11.8}$  LAS, and C<sub>12</sub>-C<sub>15</sub> methyl branched alkyl sulfates.

Other anionic detergent compounds herein include the sodium  $C_{10-18}$  alkyl glyceryl ether sulfonates, especially those ethers of higher alcohols derived from tallow and coconut oil; sodium coconut oil fatty acid monoglyceride sulfonates and sulfates; and sodium or potassium salts of alkyl phenol ethylene oxide ether sulfate containing 1 to 10 units of ethylene oxide per molecule and wherein the alkyl groups contain 8 to 12 carbon atoms.

5

10

20

25

1時 2000 1000 12時間 2012 14時時間 21

近代の大きなでは、10mmの大きのでは、10mmの大きないのであった。 は、

Other useful anionic detergent compounds herein include the water-soluble salts or esters of alpha-sulfonated fatty acids containing from 6 to 20 carbon atoms in the fatty acid group and from 1 to 10 carbon atoms in the ester group; water-soluble salts of 2-acyloxy-alkane-1-sulfonic acids containing from 2 to 9 carbon atoms in the acyl group and from 9 to 23 carbon atoms in the alkane moiety; alkyl ether sulfates containing from 10 to 18, especially 12 to 16, carbon atoms in the alkyl group and from 1 to 12, especially 1 to 6, more especially 1 to 4 moles of ethylene oxide; water-soluble salts of olefin sulfonates containing from 12. to 24, preferably 14 to 16, carbon atoms, especially those made by reaction with sulfur trioxide followed by neutralization under conditions such that any sultones present are hydrolyzed to the corresponding hydroxy alkane sulfonates; water-soluble salts of paraffin sulfonates containing from 8 to 24, especially 14 to 18 carbon atoms, and beta-alkyloxy alkane sulfonates containing from 1 to 3 carbon atoms in the alkyl group and from 8 to 20 carbon atoms in the alkane moiety.

The alkane chains of the foregoing non-soap anionic surfactants can be derived from natural sources such as coconut oil or tallow, or can be made synthetically as for example using the Ziegler or Oxo processes. Water solubility can be achieved by using alkali metal, ammonium or alkanolammonium cations; sodium is preferred.

Suitable fatty acid soaps can be selected from the ordinary alkali metal (sodium, potassium), ammonium, and alkylolammonium salts of higher fatty acids containing from 8 to 24, preferably from 10 to 22 and especially from 16 to 22 carbon atoms in the alkyl chain. Suitable fatty acids can be obtained from natural sources such as, for instance, from soybean oil, castor oil, tallow, whale and fish oils, grease, lard and mixtures thereof). The fatty acids also can be synthetically prepared (e.g., by the oxidation of petroleum, or by hydrogenation of carbon monoxide by the Fischer-Tropsch process). Resin acids are suitable such as rosin and those resin acids in tall oil. Naphthenic acids are

3

1. 1. 1916年,1916年,1916年,1916年,1918年

5

10

15

20

25

30

ķ,

also suitable. Sodium and potassium soaps can be made by direct saponification of the fats and oils or by the neutralization of the free fatty acids which are prepared in a separate manufacturing process. Particularly useful are the sodium and potassium salts of the mixtures of fatty acids derived from tallow and hydrogenated fish oil.

In preferred surfactant systems the anionic surfactants should be nonethoxylated, as such surfactants show superior grease and oily soil removal capability. Preferably also the surfactants are of the sulfonate or sulfate type and mixtures of anionic surfactants are particularly suitable herein, especially mixtures of sulfonate and sulfate surfactants in a weight ratio of from 10:1 to 1:5, preferably from 5:1 to 1:1.5, more preferably from 5:1 to 1:1. Especially preferred is a mixture of an alkyl benzene sulfonate having from 9 to 15, especially 11 to 13 carbon atoms in the alkyl radical, the cation being an alkali metal, preferably sodium; and an alkyl sulfate having from 10 to 20, preferably 12 to 18 carbon atoms in the alkyl radical.

Nonionic surfactants useful in the present invention are condensates of ethylene oxide with a hydrophobic moiety to provide a surfactant having an average hydrophilic-lipophilic balance (HLB) in the range from 9 to 15, preferably from 9.5 to 13.5, more preferably from 10 to 12.5. The hydrophobic moiety may be alliphatic or aromatic in nature and the length of the polyoxyethylene group which is condensed with any particular hydrophobic group can be readily adjusted to yield a water-soluble compound having the desired degree of balance between hydrophilic and hydrophobic elements.

Examples of suitable nonionic surfactants include:

1. The polyethylene oxide condensates of alkyl phenol, e.g., the condensation products of alkyl phenols having an alkyl group containing from 6 to 12 carbon atoms in either a straight chain or branched chain configuration, with ethylene oxide, the said ethylene oxide being present in amounts equal to 3 to 20,

5

10

15

20

25

30

Ė,

The season of th

preferably 5 to 14 moles of ethylene oxide per mole of alkyl phenol. The alkyl substitutent in such compounds may be derived, for example, from polymerized propylene, di-isobutylene, octene and nonene. Other examples include dodecylphenol condensed with 9 moles of ethylene oxide per mole of phenol; dinonylphenol condensed with 11 moles of ethylene oxide per mole of phenol; nonylphenol and di-isooctylphenol condensed with 13 moles of ethylene oxide.

- The condensation product of primary or secondary 2. aliphatic alcohols having from 8 to 24 carbon atoms, in either straight chain or branched chain configuration, with from 2 to 15 moles, preferably 2 to 9 moles of ethylene oxide per mole of alcohol. Preferably, the aliphatic alcohol comprises between 9 to 18 carbon atoms and is ethoxylated with between 2 and 9, desirably between 3 and 8 moles of ethylene oxide per mole of aliphatic The preferred surfactants are prepared from primary alcohol. alcohols which are straight chain (such as those derived from natural fats or prepared by the Ziegler process from ethylene, e.g., myristyl, cetyl, stearyl alcohols). Specific examples of nonionic surfactants falling within the scope of the invention include the condensation products of coconut alcohol with an average of between 5 to 12 moles of ethylene oxide per mole of alcohol, the coconut alkyl portion having from 10 to 14 carbon atoms, and the condensation products of tallow alcohol with an average of between 7 to 12 moles of ethylene oxide per mole of alcohol, the tallow portion comprising essentially between 16 and Secondary linear alkyl ethoxylates are also 22 carbon atoms. suitable in the present compositions, especially those ethoxylates of the Tergitol series having from 9 to 15 carbon atoms in the alkyl group and up to 11, especially from 3 to 9, ethoxy residues per molecule, Tergitol being a trade name of Union Carbide Corp.
  - 3. The compounds formed by condensing ethylene oxide with a hydrophobic base formed by the condensation of propylene oxide with propylene glycol. The molecular weight of the hydrophobic portion generally falls in the range of 1500 to 1800. Such

1

このではないのでは、1977年

5

10

15

20

25

30

synthetic nonionic detergents are available on the market under the trade name of "Pluronic" supplied by Wyandotte Chemicals Corporation.

Especially preferred nonionic surfactants for use herein are the  $\rm C_9^{-C}_{15}$  primary alcohol ethoxylates containing 3 to 8 moles of ethylene oxide per mole of alcohol, particularly the  $\rm C_{12}^{-C}_{15}$  primary alcohols containing 6 to 8 moles of ethylene oxide per mole of alcohol.

Preferred compositions, in accordance with the invention,

10 employ a ratio of anionic surfactant to glyceryl ester of from

0.25:1 to 4:1, preferably 0.5:1 to 2:1,

and most preferably 0.67:1 to 1.5:1.

A particularly desirable additional component of detergent compositions in accordance with the invention is a sequestering agent, present in an amount of from 0 to 60%, more usually from 5 to 50% by weight of the composition. The sequestering agents can be either organic or inorganic in form and may be water-soluble or water-insoluble in character.

Suitable inorganic builder salts include orthophosphates, pyrophosphates, tripolyphosphates and the higher polymeric glassy phosphates, silicates, carbonates, and the water-insoluble crystalline aluminosilicates such as hydrated Zeolite A, X or P.

Organic sequestering agents that can be incorporated include the aminocarboxylates such as the salts of nitrilotriacetic acid (NTA), ethylenediaminetetra acetic acid (EDTA) and diethylenetriaminepenta acetic acid (DETPA) and the methylene phosphonate analogues of these materials NTMP, EDTMP and DETPMP, as well as the salts of polycarboxylic acids such as lactic acid, glycollic acid and ether derivatives thereof as disclosed in Belgian Pat. Nos. 821,368, 821,369 and 821,370; succinic acid, malonic acid, (ethylenedioxy) diacetic acid, maleic acid, diglycollic acid, tartaric acid, tartronic acid and fumaric acid, citric acid, aconitic acid, citraconic acid, carboxymethyloxysuccinic acid, lactoxysuccinic acid, and 2-oxy-1,1,3-propane tri-carboxylic acid; oxydisuccinic acid, 1,1,2,2-ethane tetracarboxylic acid, 1,1,3,3-

5

15

20

25

30

35

第二日の西川田、このこの日の日本の日、日本の日

三、一直被衛衛衛門以上衛門一次 海路衛門 人名斯

propane tetracarboxylic acid and 1,1,2,3-propane tetracarboxylic acid; cyclo-pentane-cis, cis, cis-tetracarboxylic acid; cyclo-pentadienide pentacarboxylic acid, 2,3,4,5-tetrahydrofuran-cis, cis, cis-tetracarboxylic acid, 2,5-tetrahydrofuran-cis-dicarboxylic acid, 1,2,3,4,5,6-hexane-hexacarboxylic acid, mellitic acid, pyromellitic acid and the phthalic acid derivatives disclosed in U.S. Pat. No. 3,920,569, issued November 18, 1975.

The above-described inorganic and organic sequestering agents can be used in combination in order to ensure sequestration of each metal ion that influences detergency. An example of such a combination would be the use of an insoluble zeolite primarily for calcium, a polycarboxylate primarily for magnesium, and an amino polyphosphonate primarily for heavy metal ions.

## Additional Optional Components

The compositions of the present invention can be supplemented by a wide range of additional optional components.

A principal optional component which is a highly preferred ingredient of the compositions is an inorganic peroxygen bleach of the perhydrate type, defined for the purposes of this invention as having hydrogen peroxide associated with the molecule. Alkali metal perborates, percarbonates, persilicates and perpyrophosphates are examples of such bleaches. They are normally included at levels of from 5% to 35% by weight, preferably from 15% to 25% by weight of the composition.

Preferred perhydrates are sodium perborate mono- and tetrahydrate and sodium percarbonate. Sodium perborate is the most preferred perhydrate as the addition of this material to compositions according to the invention provides an enhancement of their grease and oily soil removal capability.

A highly preferred optional component of compositions in accordance with the invention is an organic peroxyacid bleach precursor, a so-called bleach activator. Compounds of this type are well known in the detergent art. They react with inorganic peroxygen bleaches such as described above to produce peroxy-

30

5

10

15

20

25

1. 海上的影響的海水,在原始的最初發展,但一点一点

acids which are more powerful bleaches than the inorganic peroxygen compounds. The inorganic peroxygen compound can be introduced into the wash solution as part of a conventional laundry detergent or it can be introduced as part of the detergent/emulsifier composition of the present invention, along with the organic peoxyacid bleach precursor. Highly preferred precursors include tetraacetyl ethylene diamine, tetra acetyl methylene diamine, tetraacetylglycouril, sodium p-acetoxybenzene sulfonate, sodium p- $C_7$ - $C_9$  acyloxybenzene sulfonate, penta acetyl glucose and octa acetyl lactose. The precursors are incorporated so as to provide from 5 to 50 ppm of available oxygen from the peroxy acid in the wash liquor and normally are present in an amount so as to provide an inorganic peroxygen compound to precursor molar ratio of at least 1.5:1, preferably at least 2:1 and normally in the range from 3:1 to 12:1.

Rather than use organic peroxy acid bleach precursors, which depend on reaction with an inorganic peroxygen compound, organic peroxyacid bleaches per se can be used in the compositions herein. Examples of such bleaches are perlauric acid, diperoxydodecanedioic acid, diperoxyazelaic acid, perphthalic acid and peroxymyristic acid.

Soil suspending and antiredeposition agents are also preferred components of the compositions of the invention at levels of from 0.1% to 10% by weight. Methyl cellulose and its derivatives such as water-soluble salts of carboxymethyl-cellulose, carboxyhydroxymethyl cellulose and polyethylene glycols having a molecular weight of 400 to 10,000 are common components of the present invention.

Enzymes in minor amounts are conventional ingredients of the compositions, those suitable for use including the materials discussed in U.S. Pat. Nos. 3,519,570, McCarty, issued July 7, 1970, and 3,553,139, McCarty et al., issued January 5, 1971.

Anionic fluorescent brightening agents are well-known ingredients suitable for use in the compositions herein at levels of from

30

3

The Mandale of the control of the control of the

5

10

15

about 0.05% to about 4%. Examples are disodium 4,4'-bis-(2-di-ethanolamino-4-anilino-s-triazin-6-ylamino)stilbene-2:2'-disulfonate and disodium 4,4'-bis-(2-morpholino-4-anilino-s-triazin-6-ylamino-stilbene-2:2'-disulfonate.

The present compositions also can contain suds regulating components in an amount of from 0.05% to 3%. Preferred are microcrystalline waxes having a melting point in the range from 35°C to 115°C and saponification value of less than 100. The microcrystalline waxes are substantially water-insoluble, but are water-dispersible in the presence of organic surfactants. Preferred microcrystalline waxes having a melting point from 65°C to 100°C, a molecular weight in the range from 400-1000; and a penetration value of at least 6, measured at 77°C by ASTM-D1321. Suitable examples of the above waxes include microcrystalline and oxidized micro-crystalline petrolatum waxes; Fischer-Tropsch and oxidized Fischer-Tropsch waxes; ozokerite; ceresin; montan wax; beeswax, candelilla; and carnauba wax.

Where the formulations are particulate in nature anticaking agents such as sodium sulfosuccinate or sodium benzoate can be included in varying amounts as desired. Aesthetic ingredients such as dyes, pigments, photo activated bleaches such as triand tetra-sulfonated zinc phthalo cyanine, and perfumes are also normal components of the compositions.

Detergent compositions, in accordance with the invention, can be solid or liquid and where they are solid can take any of the conventional forms, e.g., spray dried or agglomerated particles, bars or tablets. The glyceryl esters can be incorporated into spray dried granular compositions either with the components forming the spray dried base powder or as a sprayed or prilled additive to the base granules. In general, addition to the spray dried base powder is preferred.

According to another aspect of the invention, aqueous laundry liquors are provided comprising from 100 to 25,000 ppm of the compositions as hereinbefore described and such liquors can

30

5

10

20

25

THE THE PROPERTY OF THE PROPER

be formed in the conventional manner by direct dissolution of the compositions into water to form the wash solution. However, in accordance with a further aspect of the invention, the liquors can also be formed by the addition of a laundry additive product incorporating some of the components of the present invention to a previously formed wash liquor containing the remainder.

Such laundry additive products can take a variety of physical forms, viz., liquids, particulate solids, tablets, pouches or sachets or impregnated sheet substrates. Typically, but not invariably, the aqueous wash liquor will already contain an anionic surfactant and possibly also a sequestering agent and the additive product will comprise the glyceryl ester emulsifier system of the present invention. In certain preferred executions, additive products may themselves constitute compositions containing the emulsifier system and other materials, designed to be added to detergent liquors to provide a boost in overall detergency performance in addition to the improved oily soil removal provided by the emulsifier system.

Additive products in accordance with this aspect of the present invention may comprise one or more of the components of the composition in combination with a carrier such as a compatible particulate substrate, a flexible non particulate substrate or a container. Examples of compatible particulate substrates include inert materials such as clays and other aluminosilicates including zeolites both natural and synthetic in origin. Other compatible particulate carrier materials include hydratable inorganic salts such as phosphates, carbonates and sulfates.

Additive products enclosed in bags or containers are manufactured such that the containers prevent egress of their contents when dry but are adapted to release their contents on immersion in an agueous solution.

A convenient execution of this form of the additive product comprises a particulate solid composition enclosed in a container. Usually the container will be flexible, such as a bag or pouch.

30

Ž,

在中心,一个一个时间的时间,但是是是我们的时间,也是一个一个时间的时间,我们也是我们的时候,我们是我们的时候,我们们们们们们们们们们们们们们们们们们们们们们们们

5

10

15

20

The bag may be of fibrous construction coated with a water-impermeable protective material so as to retain the contents, such as is disclosed in U.S. Pat. No. 4,374,747, issued February 22, 1983. Alternatively, it may be formed of a water-insoluble synthetic polymeric material provided with an edge seal or closure designed to rupture in aqueous media as disclosed in European published Patent Application Nos. 0011500, published May 28, 1980, 0011501, published May 28, 1980, and 0011968, published June 11, 1980, and U.S. Pat. No. 4,348,293, issued September 7, 1982. A convenient form of water frangible closure comprises a water soluble adhesive disposed along and sealing one edge of a pouch formed of a water impermeable polymeric film such as polyethylene or polypropylene.

An alternative form of the additive product comprises a composition in water releasable combination with a nonparticulate flexible substrate in a weight ratio of 1:10 to 30:1. Laundry additive products in this form, suitable substrates for such products and procedures for preparing such products, are disclosed in U.S. Pat. No. 4,220,562, Spadini et al., issued September 2, 1980, incorporated by reference herein.

The invention will be illustrated by the following examples.

#### **EXAMPLE 1**

35 gms glyceryl monolaurate (GML) and 15 gms glyceryl monocaprate (GMC) both having 90% minimum monoester content, were melted and stirred at 65°C to 70°C for approximately five minutes. The melt was poured onto a cold surface upon which it solidified. The resulting solid was ground to a powder and passed through a 16 mesh (U.S. Standard) screen. The result is a 70:30 blend of GML/GMC.

EXAMPLE II

A detergent composition having the following formula was prepared by dry-mixing the granular GML/GMC composition of Example I with the other materials of the composition in the proportions indicated.

35

30

10

15

20

;28.6% GML/GMC of Example I

- 21.50%  $C_{11}$ - $C_{12}$  linear alkyl benzene sulfonate ( $C_{11}$ - $C_{12}$  LAS, Calsoft F-90 $^{\rm R}$ , powdered form)
- 21.50% C<sub>12</sub>-C<sub>14</sub> alkylethoxylate (2.25) sulfated (100% active dried paste)
- 22.30% Sodium tripolyphosphate (anhydrous powder)
- 2.05% Flourescent Whitening Agent
- 4.05% Enzyme Granulate (protease/amylase T-Granulate from Novo Inqustri)

This composition is formulated to be used at about 1300 ppm in a typical 17 gallon U.S. washing machines.

### EXAMPLE III

The detergent composition described in Example II was utilized as a laundry additive in addition to the normal laundry detergent in a performance test. The test was conducted in a 2 gallon automatic miniwasher. The two treatments were, TIDER detergent alone at recommended usage of 1500 ppm (11.3 gms), and  $\mathsf{TIDE}^R$  at recommended usage plus the detergent composition described in Example II at 1320 ppm (2.64 gms). The water was set at 95°F temperature and 7 grains/gallon water hardness. 290-300 gms. of fabrics soiled with a laboratory-simulated body soil were added to each wash, simulating a real laundry load. Performance was measured by visual comparison by expert graders of cotton, and polyester/cotton blend swatches which were prepared in an identical manner for each treatment. In this test, the detergent plus test composition gave better overall stain removal performance than detergent alone, particularly on grease/oil stains such as bacon grease, makeup, and facial body soil. The results are reported in Table 1. The data in the table represent the difference in average panel score (visual grading) between the detergent and the detergent plus additive. Indicates an advantage for detergent + additive.

30

5

10

15

20

TABLE 1
STAIN REMOVAL RESULTS

# Detergent vs. Detergent + Additive

	Stain	Fabric	Panel Score
5	Spaghetti sauce	*P/C 50/50	+1.7 (s)**
	Dirty motor oil	Cotton	+0.2
	.Bacon grease	Cotton	+1.3 (s)
	Make-up - oil based	P/C 65/35	+3.3 (s)
	Facial body soil	P/C 65/35	+1.2 (s)
10	Tea	P/C 65/35	+0.2
	Grape	P/C 65/35	+0.5
	Blood	Cotton	+2.0 (s)
	Grass	Cotton	+1.8 (s)
	Chocolate	Cotton	-0.8
15	Gravy	Cotton	+0.1
	Clay	Cotton	-0.1

\*P/C = polyester/cotton blend

\*\*(s) = Statistically sign. at 90% conf.

# 20 EXAMPLE IV

22.5 gms glyceryl monolaurate and 9.65 gms glyceryl monocaprate were melted and stirred at 65°-70°C. To this liquid was added the following solids: 24.10 gms C<sub>11</sub>-C<sub>12</sub> LAS, 24.95 gms of AE 2.25S, 25.0 gms sodium tripolyphosphate, 2.3 gms Fluorescent Whitening Agent #27, and 4.5 gms protease/amylase enzyme. This mixture has the same formula as the composition in Example II. The temperature was maintained at 65°-70°C, and the mixture was stirred to produce a fairly uniform slurry. A pre-weighed 9"x14" nonwoven rayon substrate containing 1-2 mm diameter aperatures was placed on a flat heated steel plate and 23 gms of the slurry was poured onto the substrate and spread to obtain an even coating. The substrate was removed, allowed to cool in ambient, low humidity air in order to permit the active to solidify, and then it was weighed. The substrate was then returned to the hot plate, additional slurry was added, followed by cooling and

25

30

35

A CONTRACT TOWNS AND CONTRACT OF THE CONTRACT OF

weighing. This process was repeated until 22.4 gms of the composition was bound to the substrate.

This article is intended for use as a laundry additive by adding to a laundry detergent solution in a typical 17 gallon capacity washing machine.

10

15. 有量,每12.000 mmense 中心12. 5.

A CONTRACTOR OF THE CONTRACTOR

5

15

20

25

30

#### CLAIMS

- 1. A composition of matter comprising monolaurin and monocaprin in a weight ratio of from 80:20 to 50:50 monolaurin:monocaprin.
- A composition according to claim 1 wherein the ratio of
   monolaurin:monocaprin is from 80:20 to 70:30.
  - 3. A detergent composition according to either one of claims 1 or 2 comprising:
  - (a) From 5% to 60% of an emulsifier system comprising monoplaurin and monocaprin
- 10 (b) From 5% to 60% of a synthetic detergent selected from anionic and ethoxylated nonionic detergents, and
  - (c) From 0% to 60% of a sequestering agent.
- A composition according to claim 3 wherein Component (a) is present in an amount of from 20% to 45%, Component (b) is
   present at from 25% to 50%, and Component (c) is present at from 5% to 50%.
  - 5. A composition according to either one of claims 3 or 4 wherein the amount of Component (a) is from 25% to 35%, and the amount of Component (b) is from 35% to 45%.



# **EUROPEAN SEARCH REPORT**

0131393

Application number

EP 84 30 3972

	DOCUMENTS CON	SIDERED TO	BE RELEVAN	 T	٦	
Category	Citation of document w			Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. CI. <sup>3</sup> )	
D,A	US-A-4 000 340 al.) * Claims 1,2 *	(A.P. MUI	RPHY et		C 11 D 3/20 D 06 M 13/16	
A	DE-A-2 701 266	 (UNILEVER	R N.V.)			
	* Page 14, exa *	mple 1, cl	laims 1-3			
A	DE-A-2 700 512 GAMBLE CO.) * Claims 1,5 *	(PROCTER	&			
		•	٠		TECHNICAL FIELDS SEARCHED (Int. CI. 7)	
				_	C 11 D 3/00 D 06 M 13/00	
				,		
				•	;	
	The present search report has b					
Place of sompletion  Date of sompletion		tion of the search B-1984	SCHUI	TZE D		
X: particularly relevant if taken alone Y: particularly relevant if combined with another document of the same category A: technological background			after the filin D: document ci L: document ci 8: member of ti	: theory or principle underlying the Invention : earlier patent document, but published on, or after the filing date : document cited in the application : document cited for other reasons  k: member of the same patent family, corresponding document		

and the formal of the second of the second s